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TITLE : SYNTHETIC QUARTZ GLASS POWDER CONTAINING ALUMINUM, SYNTHETIC
QUARTZ GLASS FORMED BODY CONTAINING ALUMINUM, AND PRODUCTION OF
THE SAME

ABSTRACT : PROBLEM TO BE SOLVED: To suppress crystallization of a formed body when used at a high temp. by controlling the number of devitrified spots of a specified size to a specified value or smaller produced in an ingot which is obtd. by fusing in vacuum at a specified temp. when the ingot is kept under specified conditions.

SOLUTION: The synthetic quartz glass powder after produced is fused in vacuum at 1,780 to 1,800°C to obtain an ingot. When the ingot is kept at 1,630°C for 5 hours, the number of devitrified spots having $\geq 20 \mu\text{m}$ diameter produced in the ingot is specified to ≤ 10 per 50 g, preferably ≤ 5 per 50 g. The synthetic quartz glass powder contains Al so as to improve the high temp. viscosity of the final glass product, and the Al content is preferably controlled to 0.5 to 500 ppm. The synthetic quartz glass powder is preferably produced by a sol-gel method by reacting a silicon alkoxide with water to hydrolyze, gelatinizing and drying the product to obtain a silica gel, and vitrifying the gel.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[The technical field to which invention belongs] this invention relates to synthetic quartz powder suitable as an object for optics which has the process by which high temperature processing is carried out at the time of manufacture of a semiconductor field, the object for semiconductor manufacture for quartz glass especially used in a high temperature field 1000 degrees C or more, or an optical fiber, and a quartz-glass product.

[0002]

[Description of the Prior Art] In recent years, about the quartz-glass product used for a semiconductor industry field, very severe management is performed about the purity. It faces obtaining the glass of such a high grade, and although manufactured by fusing the living-rock English powder which refined and ground the natural quartz and obtained it conventionally, the high thing of cost is increasingly required also about refining technology as high integration of a semiconductor and the demand of improvement in the yield become severe. On the other hand, apart from this, although there are some which depend a silicon tetrachloride on the gaseous-phase method used as the raw material as a method of obtaining the quartz glass of a high grade, much energy is required and it has the fault that efficiency is bad. The synthetic quartz powder by the so-called sol gel process which makes silica gel by the liquid phase reaction as a method of solving these troubles by using organosilicon compounds, such as for example, a silicon alkoxide, as a raw material, and is further used as glass through processes, such as dryness, pulverization, and baking, is obtained, and there is the method of carrying out melting fabrication of this and using as a quartz-glass product.

[0003] However, there are few examples independently used for a high-temperature-service way since elevated-temperature viscosity is low as compared with that to which the quartz-glass product obtained by carrying out melting fabrication of the synthetic quartz powder generally used living-rock English powder as the raw material, for example, the crucible of the dual structure to which made the outside as the natural quartz and they made the inside synthetic quartz is produced, and the device which secures a high temperature strength is made (JP,3-40989,A). Moreover, the method of carrying out minute amount addition of the aluminum into glass is learned as the technique of raising the elevated-temperature viscosity of synthetic quartz glass itself (JP,61-236619,A, JP,3-45530,A). although the detail of the mechanism of the improvement in elevated-

temperature viscosity by aluminum addition is not yet made clear, it exists as glass modifier oxides (end group), such as Na, K, and OH, and the alumina of (1) (2) to which trap of ion to which elevated-temperature viscosity is reduced is carried out high-melting point checks a viscosity-of-glass flow -- the opinion of ** is leading

[0004]

[Problem(s) to be Solved by the Invention] Especially the crucible for silicon-single-crystal raising is used at the elevated temperature of 1400-1500 degrees C as a use of synthetic quartz glass for a long time. The continuation Czochralski method performed while throwing in raw material silicon continuously especially in recent years comes to be enforced, and the time of a crucible is turning further for a long time. Since the glass phase (amorphous) which is the form of a crucible is a metastable phase, if it holds in the above-mentioned temperature field for a long time, it tends to transfer to the crystalline substance (cristobalite) which is a stable phase. If a metal impurity etc. exists especially, this will serve as a crystalline nucleus and will promote crystallization more. during single crystal raising, when this phenomenon (generally called devitrification) arises, the crystalline-substance portion generated into the crucible wall portion will dissociate, it will mix into a silicon melt, and single crystal-ization will be blocked (UCS semiconductor base technical research meeting edit "science of silicon", 1996) Moreover, also about the work tube used for heat treatment of a silicon wafer etc., although it is low, the service temperature will cause an on-the-strength fall and breakage by the repeat of heating and cooling, since coefficient of thermal expansion differs in a crystalline substance and an amorphous portion, if devitrification arises by using it for a long time. To these troubles, synthetic quartz glass has a metal impurity content in very low level as compared with natural crystal powder, and it cannot devitrify it easily.

[0005] As mentioned above, although there is [minute amount addition of aluminum] an effect in improvement in elevated-temperature viscosity of synthetic quartz glass, on the other hand, the bird clapper became [the devitrification phenomenon] being easy to be discovered [with addition of aluminum] clear by examination of this invention persons. Planning high viscosity is performed by applying and printing aluminum on the front face of a quartz-glass product, and it is easy to devitrify the front face of a quartz-glass product by this method. That is, causing devitrification by existence of aluminum is known. To this, in order to make it distribute uniformly on molecule level in glass as addition time of aluminum, a sol gel process is adopted, and it is possible to add at the time of the hydrolysis. With each above-mentioned well-known technology about a sol gel process, the alkoxide of aluminum is actually added in the hydrolysis liquid of a silicon alkoxide.

[0006] However, when melting fabrication was carried out using the aluminum content synthetic quartz glass powder obtained in this way, the problem which many bubbles generate might arise in the done Plastic solid. Previously, this invention persons have proposed adding an aluminium compound in the raw material stage in a sol gel process in order to suppress the foaming phenomenon of aluminum content synthetic quartz glass (JP,10-287417,A, JP,10-287418,A). However, when melting fabrication was carried out using the aluminum content synthetic quartz powder with which the foaming phenomenon was suppressed in this way, many devitrification spots might occur in the done ingot. This is guessed because there is a portion in which aluminum exists by high concentration locally. That is, it was guessed that the homogeneity of distribution of

aluminum to the extent that a devitrification phenomenon can be prevented cannot be attained in addition even if it adopts the sol gel process considered that it can generally carry out uniform distribution of the raw material and adds an aluminium compound before vitrification.

[0007]
. [Means for Solving the Problem] In order that this invention persons may solve the above-mentioned technical problem, as a result of repeating examination wholeheartedly, the aluminum content synthetic quartz glass powder in the criteria that the physical-properties value evaluated by the specific evaluation method is fixed found out that generating of the devitrification phenomenon at the time of carrying out melting fabrication could be prevented. And such an aluminum content synthetic quartz glass powder also found out that it could obtain by taking specific means in a sol gel process. Namely, this invention is a synthetic quartz glass powder containing aluminum. When vacuum fusion was carried out at 1780-1800 degrees C, it considered as the ingot and it holds in 1630 degrees C for 5 hours, The aluminum content synthetic quartz glass powder characterized by the devitrification spots of the size more than 20 micrometerphi generated in this ingot being ten pieces / 50g or less, It consists in the aluminum content quartz-glass Plastic solid characterized by including the process which is 1700 degrees C or more, and holds the synthetic quartz glass powder containing aluminum 30 minutes or more. Although the mechanism by which generating of a devitrification spot is suppressed is not clear if melting fabrication of the aluminum content synthetic quartz glass powder of this invention is carried out, probably aluminum is distributing highly, and aluminum does not exist by high concentration locally from this, but it is thought that generating of a devitrification spot can be prevented.

[0008]

[Embodiments of the Invention] Hereafter, this invention is explained in detail. Most preferably as the manufacture method of an aluminum content synthetic quartz glass powder which satisfies this invention, silica gel is obtained by hydrolysis, gelling, etc. of a silicon alkoxide, and there is the method of performing by the sol gel process which vitrifies this. Although the method of obtaining silica gel here is not limited, a silicon alkoxide and water are made to react typically and there is the method of hydrolyzing, gelling, drying and making it into silica gel. Although not limited especially as a silicon alkoxide, what is the basis which can hydrolyze all the bases combined with silicon from the point that survival of the carbon to the aluminum content synthetic quartz glass powder obtained can be prevented easily is desirable, and a point to especially the tetrapod alkoxysilane that can also prevent especially survival of chlorine etc. is desirable. The oligomer which is the low-grade alkoxysilane or its low condensate of C1-C4 as tetrapod alkoxysilane is easy to hydrolyze, and desirable from a point with little carbon survival into silica gel. The tetramethoxy silane from the cheap point of especially a price is desirable.

[0009] Although these silicon alkoxide is made to react with water, and it hydrolyzes, gels and dries and being considered as silica gel, it considers as aluminum content silica gel by adding the source of aluminum. If there are about 0.5-50 ppm as an aluminum content for raising the elevated-temperature viscosity of the last glassware, even if it is enough and is added more than this, the aluminum concentration which the effect of the improvement in elevated-temperature viscosity hardly changes, and serves as an impurity to a semiconductor may be not much high, and a bird clapper's may not be desirable. On

the other hand, if there are few aluminum additions than the above-mentioned range, the viscous improvement effect that it can bear enough will become inadequate for elevated-temperature use of 1000 degrees C or more. As a content of aluminum, 0.1-100 ppm is usually 0.5-50 ppm.

[0010] Although especially the source of aluminum that can be used is not limited, an aluminium compound with a silicon alkoxide and compatibility is desirable, and it is desirable especially to specifically use the chelate compound of aluminum. It is not limited especially as a chelate compound of aluminum, for example, aluminum tris (ethyl acetonato), an aluminum monoacetyl acetate screw (ethyl acetoacetate), and aluminum tris (acetylacetonato) (it is also called aluminium acetylacetonato) are mentioned. Since near and compatibility also have a silicon alkoxide and high adding-water catabolic rate also especially in it, aluminium acetylacetonato is used suitably. Although this aluminum chelate compound was added so that it might become the above-mentioned concentration into the last glassware, it became clear by examination of this invention persons that it is desirable for obtaining the aluminum content synthetic quartz glass powder of this invention before adding water to the stage, especially silicon alkoxide before gelling as addition time. As the addition method, since the target concentration in a final product is low, aluminum concentration is opposite SiO₂ beforehand. After leaving it so that an about 80-1200 ppm thick solution may be adjusted preferably and this may be later mentioned 60-1500 ppm by conversion, it is used adding a silicon alkoxide, and the precision of aluminum concentration adjustment is [direction] good, and it is efficient.

[0011] If a adding-water decomposition reaction is started immediately after adding this aluminum chelate compound in a silicon alkoxide, now, the dryness gel obtained after gelling, pulverization, and dryness It compares with the gel reacted and obtained only by the usual silicon alkoxide. In order specific surface area is small, for a thing and a bird clapper with much foaming to be found out by this invention persons when the black foreign matter which originates in unburnt carbon at the time of baking is generated and it fuses, and to avoid this, It has proposed previously leaving the silicon alkoxide which added the aluminium compound so that it might state below for 5 hour - one day (JP,10-287417,A, JP,10-287418,A). However, in neglect of this level, although foaming was suppressed, fully not being stopped made devitrification clear. It was found out by this invention persons that it is effective for devitrification prevention to leave it at least two days or more.

[0012] Thus, although the mechanism which can prevent devitrification by leaving it for a long time is not clear, probably the following things can be considered. Although it is as above-mentioned that it is thought desirable to use an aluminum chelate compound as an aluminum content compound from the field of adding-water catabolic rate for attaining uniform mixture Although an aluminum chelate compound exists as a polymer to which some aluminum content units generally met After silicon compounds, such as tetrapod alkoxysilane, are mixed, a meeting state is released gradually. It is thought that it distributes on single molecule level, and in order to acquire the uniform dispersibility of the grade which does not cause the manifestation of a devitrification spot, what has required the long time more sharply than time to usually be predicted and get is conjectured. that is, although the solubility of an aluminum chelate compound to a silicon alkoxide is large, and thinking that it already comes out enough as uniform dispersibility of aluminum usually comes out when an aluminum chelate compound dissolves, since it

will dissolve promptly, if it adds. Against such prediction, it is not enough for devitrification prevention just to have made it dissolve, and it is conjectured whether to have contributed to devitrification prevention that an aluminium compound is understood by after an appropriate time an added water part through a certain aging. Although the mechanism of this aging is not clear, since it is thought that change is promoted by heating, although the neglect on the 2nd or more is appropriate at least at a room temperature, it is thought under heating that a short time is also more enough.

[0013] If it is not left or the below-mentioned adding-water decomposition reaction is carried out in a stage with inadequate neglect, the devitrification Ming spot in the Plastic solid finally obtained will increase. It will be a book, if the solubility to the silicon alkoxide of an aluminum chelate compound is large, and it will dissolve promptly if it adds and is generally only left after addition, although it not only leaves it, but you may perform mixture, churning, heating, etc. at the time of neglect. That is, although it is unknown whether it is what is depended on what mechanism, the aluminum content synthetic quartz glass powder of this invention and also the aluminum content quartz-glass Plastic solid of this invention can be obtained by satisfying the easy requirements of setting without adding the water which hydrolysis will take after [addition] two days or more also unexpectedly.

[0014] Thus, when the aluminum concentration in the silicon alkoxide which carried out specific time neglect is over the target concentration of the last glassware, after adding a silicon alkoxide according to the concentration of final aluminum and diluting aluminum concentration, water is added and a adding-water decomposition reaction is started. The effect is not barred, even if it makes a adding-water decomposition reaction start immediately, after diluting with this silicon alkoxide. In addition, as an amount of the water to add, it is usually below the 10 time equivalent more than the 1 time equivalent required for a reaction. Moreover, you may mix organic solvents, such as alcohols which have compatibility in both an alkoxide and water if needed at the time of hydrolysis, and ether and ketones. As alcohol, diethylether etc. is mentioned as ether and a methanol, ethanol, propanol, etc. are mentioned for an acetone etc. as ketones. However, in the case of a raw material which has the large alkoxy group (for example, methoxy machine) of adding-water catabolic rate, when reaction mixture will be in a uniform state before gelling since the alkoxy group combined with the alkoxide along with advance of a adding-water decomposition reaction separates as alcohol, even if it does not add alcohol, it can operate without trouble in practice. In addition, though natural, let these raw material liquid be a high grade altogether.

[0015] A adding-water decomposition reaction is mostly ended after the time of the uniform solution of an alkoxide and water being formed. Subsequently, what is necessary is just to put until a solution gels, after becoming a uniform phase. Although a adding-water decomposition reaction and the conditions of gelling change with raw materials used, it is usually 20 minutes - about 10 hours under the flow and pressure requirement of an ordinary pressure under the temperature of 20-80 degrees C. Although gel can be immediately obtained if it heats in order to make a hydrolyzate gel, since it gels in several hours even if it leaves it in ordinary temperature, the gelation time can be adjusted by adjusting the temperature of heating. Although the specific surface area of gel is not limited, its specific surface area measured by the nitrogen adsorption process is effective for suppressing foaming in the quartz-glass Plastic solid considered to originate in

residual carbon more than $400\text{m}^2/\text{g}$ by considering as the thing 500 more m^2/g or more. Thus, the obtained gel is wet gel which usually contains moisture 70% of the weight or more.

[0016] In this way, the obtained silica gel is subdivided by trituration etc. if needed, and it considers as silica gel powder. Moreover, silica gel is dried prior to baking generally mentioned later. In this case, after you may dry after subdividing gel, and drying, you may subdivide. Anyway, 10-1000 micrometers of particle size after dryness subdivide so that it may be preferably set to 100-600 micrometers, and they sets a mean particle diameter to 150-300 micrometers. The obtained wet gel is adjusted to arbitrary grain size by trituration. What is necessary is just to decide the optimal grain size of wet gel in consideration of a contracted part by dryness and baking, in order to acquire the particle size distribution of the final product made into the purpose at this time. Usually, it is adjusted to the grain size of the range of 50-1000 micrometers as a final product.

[0017] After dryness, the ground wet gel is calcinated and let it be synthetic quartz powder. Here, dryness is usually carried out at the temperature of 50-200 degrees C, removes free water and the isolation methanol in wet gel, and is taken as dry gel. Although under an ordinary pressure or reduced pressure and heating temperature changes also with conditions, it is usually 50-200 degrees C. [dryness] Moreover, both a batch and continuation can perform operation. The grade of dryness is usually performed to 1 - 30 % of the weight by the content of water. Thus, although heat the obtained dryness silica gel powder in a 1000-1300-degree C temperature field, it is made to turn nonporous finally and is used as synthetic quartz powder, before sealing of gel produces the carbon component which lengthens the holding time in a 300-600-degree C temperature field, and originates in adsorption alcohol or an alkoxy group, when the fully removed direction suppresses foaming at the time of melting, it is desirable.

[0018] It is important for this to fully remove the carbon component (for most to be an alkoxy group) which remains in dry gel, before nonporous-ization of gel advances in order to suppress foaming at the time of obtained synthetic quartz powder melting. This sake, It is effective to perform the device which makes temperature up inclination loose or prepares maintenance in oxygen content atmosphere in the 300-600-degree C temperature field which is especially the desorption temperature of an alkoxy group. However, even if it devises the above baking conditions in this case as the specific surface area of the silica gel powder before baking is under $400\text{m}^2/\text{g}$, it becomes difficult to suppress generating of a black foreign matter in the satisfactory range. Therefore, in the stage of the silica gel powder before this baking, it is desirable to carry out to more than $500\text{m}^2/\text{g}$ preferably more than at least $400\text{m}^2/\text{g}$. Thus, it can attain by leaving an aluminium compound and a silicon alkoxide mixed solution as a method of manufacturing gel with a big specific surface area, for example, as mentioned above.

[0019] the quartz glass with which the use of the synthetic quartz glass powder finally obtained on the other hand is used in an elevated-temperature field 1000 degrees C or more -- when it is a member, since the method of a low is high, its amount of silanol-group survival after baking is [elevated-temperature viscosity] desirable, and a thing 60 ppm or less is usually preferably desirable 100 ppm or less as residual silanol concentration In order to lower silanol concentration efficiently, the dew-point of a controlled atmosphere has a good method of a low, and it is [-20 degrees C or less of dew-points] desirable that it is -40 degrees C or less preferably. Since the diffusion rate

of a silanol becomes [the higher one] quick as a baking maximum temperature, although the thing of the target silanol concentration can be obtained more in a short time, it usually carries out in 1000-1300 degrees C from sintering of particles, the heat-resistant problem of a baking container, etc.

[0020] Moreover, as an atmosphere of baking, considering as oxygen content atmosphere is desirable up to near at least 600 degree C. Furthermore, in order to obtain the low quartz-glass powder of the content of a silanol group, on the low gas of a moisture content, and a concrete target, it calcinates -20 degrees C or less of dew-points, introducing gas of -40 degrees C or less of dew-points in a system preferably. About the gas introduced at this time, what was beforehand filtered with the filter is used so that impurities, such as metal dust, may not enter in a system. As capacity of a filter, 1 micrometers or more of things of a performance which can remove a particle 0.5 micrometers or more preferably are chosen. Moreover, as the quality of the material of a filter, the thing made from polypropylene and Teflon is suitably used for the organic macromolecule system of a high grade, and a concrete target. Even if it is an organic macromolecule system, the high thing of what has a high ash content especially alkali, and an alkaline-earth system metal content is not desirable. The holding time in the maximum temperature of baking is usually the range of 10-100Hr, although the holding times also differ naturally, since the reduction speed of a silanol group changes with temperature.

[0021] Although the aluminum content synthetic quartz glass powder of this invention can be obtained by the method described above, it is not limited to this. It is a synthetic quartz glass powder containing aluminum, and when vacuum fusion is carried out at 1780-1800 degrees C, it considers as an ingot and this is held at 1630 degrees C for 5 hours, the devitrification spots of the size more than 20 micrometerphi generated in this ingot should just be ten pieces / thing 50g or less. This devitrification Ming spot is measured as follows. That is, 50g of synthetic quartz glass powders is put into heat-resistant containers, such as a crucible, and at the temperature of 1780-1800 degrees C, it vacuum-fuses, and cools, and an ingot is produced. This ingot is heated in the range of 1630 degrees C, and is held for 5 hours, and it cools. Although the about dozens-1mm thing which changed spherically white may be observed when the inside of this ingot is observed, this is the "devitrification spot" which poses a problem. As a result of this invention persons' repeating research, between the number of manifestations of this "devitrification spot", and crystallization which casts to a final product and generates this at the time of use It turns out that there is big correlation. the devitrification spot of the size more than 20 micrometerphi Ten pieces / 50g or less, Preferably, if the crucible for silicon-single-crystal raising etc. manufactures the quartz-glass member used at an elevated temperature using five pieces / aluminum content synthetic quartz glass powder which is two pieces / 50g or less especially preferably 50g or less It became clear that the crystallization phenomenon of glass which poses a problem does not arise even if it uses this quartz-glass member under the usual conditions. And it succeeded in obtaining conventionally what actually suppressed the manifestation of a devitrification spot to the low extremely in the aluminum content synthetic quartz glass powder which a devitrification spot tended to discover.

[0022] The size of a devitrification spot can be expanded and measured using an optical microscope etc. If the configuration of a devitrification spot is a globular form mostly, a

size will be specified with the diameter. Although the configuration of a devitrification spot may not necessarily be a globular form, a major axis prescribes a size in that case. In addition, although it is thought that the mechanism which a devitrification spot generates is complicated and it is not yet clear, it is possible that the existence by the local high concentration of aluminum works as nucleation matter, and produces crystallization at the time of elevated-temperature use. And existence of the aluminum in a gestalt which is committed as nucleation matter by the means of prolonged neglect in a raw material stage by the above-mentioned method is suppressed, and it is surmised that it is one effective for devitrification prevention. Thus, since it is guessed that the existence by high concentration with local aluminum causes devitrification, in order to prevent the existence by the local high concentration of aluminum, after vitrifying, the processing which makes aluminum high-decentralize in synthetic quartz glass is effective by holding above 1700 degrees C. By such processing, the devitrification spot in the inside of an aluminum content quartz-glass Plastic solid can be reduced. In this case, as mentioned above, it is more desirable from the homogeneity of the distributed state of aluminum, and a viewpoint of devitrification prevention to add aluminum in the stage before vitrification further using what was obtained by hydrolysis of tetrapod alkoxysilane, dryness, and baking as an aluminum content synthetic quartz glass powder.

[0023] Thus, when a high thing can be obtained also about the thermal resistance by [various kinds of] carrying out a glass member as a quartz-glass Plastic solid as compared with the conventional aluminum additive-free synthetic quartz glass member by carrying out melting fabrication using the obtained aluminum content synthetic quartz powder, crystallization in a Plastic solid can be prevented. more -- concrete -- the various fabricating methods, such as a vacuum fusion method of the arc melt method and a Verneuil method, -- the quartz glass for semiconductor manufacture, such as CHUPU of the crucible for silicon-single-crystal raising, and a diffusion furnace, and a fixture, -- it is suitable especially as high grade quartz-glass members as which a high temperature strength is required, such as a member Of course, even if it uses an optical fiber, IC sealing agent, etc. for uses other than the use in an elevated temperature, it does not interfere. Hereafter, an example explains still in detail.

[0024] (Example 1) aluminium acetylacetonato powder: -- 12.0g -- tetramethoxy silane: - it was made to dissolve in 1000g, put into the 1l. plastic container, and was left at the room temperature in the state of sealing for 48 hours (this is called Solution A) Next, preparing and agitating tetramethoxy silane:2000g and the A:4g of the above-mentioned solutions in a 5l. separable flask with a jacket, water:1184g was added and the adding-water decomposition reaction was made to start. In addition, jacket temperature of this reaction time was made into 50 degrees C. At first, in the state of the emulsion, although the solution was cloudy, it became a transparent uniform phase after about 20 minutes. Furthermore, reaction mixture was moved and changed into the 5l. plastic container from the reaction container lower part after 3 minutes, and gentle placement and the gelling of were done. What was gelled was ground passing the nylon network of 900 micrometers of diameters of opening, the vacuum drying was performed at 200 degrees C for 10 hours, it considered as dry gel powder and the classification was carried out to the range of 100-500 micrometers. Then, this dry gel powder was put into the 2l. square shape quartz-glass container, and it set to the electric furnace. It calcinated in 2 hours by carrying out at a temperature up and 500 degrees C to 500 degrees C in 4 hours, and carrying out at a

temperature up and 1200 degrees C to 10-hour maintenance and 1200 degrees C for 40 hours, circulating dehumidifying air of -40 degrees C of dew-points in a preparation fine-particles front face. Moreover, as for the aluminum content in fine particles, it was checked as a result of analysis 4.8 ppm and that aluminum is mostly doped as calculated value.

[0025] The aluminum content synthetic quartz glass powder which is the obtained fine particles was taken out, among these 50g was put into the crucible made from a graphite, vacuum fusion was carried out at 1800 degrees C, and the ingot was produced. 5Hr maintenance of this ingot was carried out at 1630 degrees C in Ar gas atmosphere, and when the interior of the obtained ingot was observed under the microscope after cooling, the devitrification spot of the size more than 20 micrometerphi was not seen.

[0026] (Example 1 of comparison) The same operation as an example 1 was performed except having changed the neglect time in the sealing state after making it dissolve in a tetramethoxy silane and moving aluminium acetylacetonato powder to a plastic container in 48 hours, and having considered as 24 hours. 100 devitrification spots were observed among the 50g ingot.

[0027]

[Effect of the Invention] The aluminum content quartz-glass Plastic solid by which crystallization was suppressed by this invention at the time of elevated-temperature use can be obtained.

[Translation done.]

CLAIMS

[Claim(s)]

[Claim 1] The aluminum content synthetic quartz glass powder characterized by being a synthetic quartz glass powder containing aluminum, and the devitrification spots of the size more than 20 micrometerphi generated in this ingot being ten pieces / 50g or less when vacuum fusion is carried out at 1780-1800 degrees C, it considers as an ingot and this is held in 1630 degrees C for 5 hours.

[Claim 2] The aluminum content synthetic quartz glass powder according to claim 1 whose devitrification spots of the size more than 20 micrometerphi generated in an ingot are five pieces / 50g or less.

[Claim 3] The aluminum content synthetic quartz glass powder according to claim 1 or 2 whose aluminum concentration is 0.5-50 ppm.

[Claim 4] Aluminum content silica gel powder whose devitrification spots of the size more than 20 micrometerphi generated in the ingot it is the silica gel powder containing aluminum, and are ten pieces / 50g or less when calcinated, consider as an aluminum content synthetic quartz glass powder, vacuum fusion is carried out at 1780-1800 degrees C, it considers as an ingot and it holds at 1630 more degrees C for 5 hours.

[Claim 5] The aluminum content synthetic quartz glass powder which comes to calcinate aluminum content silica gel powder according to claim 4.

[Claim 6] The manufacture method of the aluminum content quartz-glass Plastic solid

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characterized by including the process which is 1700 degrees C or more, and holds the synthetic quartz glass powder containing aluminum 30 minutes or more.

[Claim 7] The manufacture method of an aluminum content quartz-glass Plastic solid according to claim 6 that the synthetic quartz glass powder containing aluminum is obtained by hydrolysis of tetrapod alkoxysilane, dryness, and baking.

[Claim 8] The manufacture method of the aluminum content quartz-glass Plastic solid characterized by carrying out melting fabrication of the aluminum content synthetic quartz glass powder according to claim 1, 2, 3, or 5.

[Claim 9] The aluminum content quartz-glass Plastic solid obtained by the manufacture method according to claim 6 to 8.

[Claim 10] The aluminum content quartz-glass Plastic solid according to claim 9 whose content of aluminum is 0.1-100 ppm.

[Translation done.]

